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Introduction

Experiments with the wetting behavior of immiscible fluids against the container below the critical temperature are being performed in the MSFC Drop Tower facility. Microgravity conditions extending up to three seconds (of the 4.5 second drop) are generated for the experiment. Specimens consist of glass cylindrical ampoules partially filled with fluid phases. How the fluids develop the meniscus geometry as well as how the fluid interfaces respond to the microgravity induced oscillations is recorded during the experiment with on-board cameras. Drops are made at various temperatures to determine the interfacial energy variation as a function of temperature.

This work originally began several years ago, but with endless delays in getting experiments performed the grant has been no-cost extended until July 1989. There are results of some value. Recent facility improvements should increase the quality of the data during the next year. During these years, the experiment has been modified, improved and refined with some initial objectives canceled due to funding and time.

MSFC Drop Tower Facility

The Experiments described here rely on the character of the gravity environment produced by the Drop Tower. Initially, the experiment package (270 kg and 1x1x1 meter dimensions) sits within the drag shield at the top of the tower (100 meters nominal). Upon release, a pressurized gas rocket thruster accelerates the (over 1000 kg) drag shield such that the experiment package floats up from the drag shield floor (about 5 cm). Microgravity conditions within the experiment begin at this point and have levels reaching 10^{-3} to 10^{-5} g. The thruster ensures the air drag doesn't influence the experiment and keeps the drag shield falling with the rate of 1 g.

The drag shield is decelerated by a catch tube which permits a controlled release of compressed air as the close fit-

ting drag shield enters the tube. The package settles on the floor and finally all comes to rest with up to a maximum of 25 g deceleration.

Experiment Summary

During the drop, accelerometer data and photographic recording of the fluid phenomena is taken for later analysis.

Most of this work has been performed with the one well documented immiscible system having a T_w, the perfluoromethylcyclohexane and isopropanol (P-I) system found by Schmidt and Moldover. The critical temperature, T_c, is 90.5 ±0.5°C, and the T_w was found to be 38.0 ±0.1°C. At temperatures above T_w, the more dense perfluoromethylcyclohexane-rich phase (with density of 1.768 g/cm at 22°C) will form a wetting layer between the vapor phase and the upper, less dense isopropanol-rich phase (of density 0.826 g/cm at 22°C). Experiments were performed in the temperature range 35 to 55 °C, encompassing the T_w.

Results

At first, the contact angle between the interfluid phases and the container wall as well as the overall meniscus shape were the most important sources of proof that a wetting transition had occurred. Due to the presence of small lateral g-forces during a drop, the meniscus shape determinations are not a sufficiently sensitive test for the critical wetting condition (in the P-I system).

An observation was made of a subtle rate of oscillation period change when the temperature was raised from below T_w to above T_w. Displacement of the mid-section or apex of the fluid interface results in a symmetrical, opposing movement of the contact line against the ampoule wall since there is no volume change. See Figure 1. A dashed line marks the meniscus profile at one-g. The sides of the graph represent the ampoule walls. The inter-fluid interfaces 'slide' up and down along the ampoule wall in response to the pulse change in g-level. The period of oscillation was found not to remain constant with each cycle. As the oscillations damped out, the velocity of the contact line on the ampoule wall also diminishes and so the dissipation from the contact line 'friction' will be considered as a function of its velocity. As it is the wetting conditions at the wall which is being tested, altering the wetting conditions should affect the oscillation rate and damping of the oscillations.

Contact Line Phenomena

The consideration now is directed to dynamic contact angles, those established during the movement of the fluid in-

terface. Experiments have shown there is a contact angle dependence on contact line velocity. The second Figure was drawn using data from a paper by Johnson (R. E. Johnson Jr., R. E. Dettre and D. A. Brandreth, " Dynamic Contact Angles and Contact Angle Hysteresis", J. Coll. and Int. Sci. 62(2) (1977) 205-212.) The static contact angle hysteresis (CAH) limits are the two values of θ where U = zero. More significantly, there is a variation in θ with velocity. Young and Davis (G. W. Young and S. H. Davis, paper submitted to J. of Fluid Mechanics, "A plate oscillating across a liquid interface: effects of contact angle hysteresis") found that both the CAH and steepening of the contact angle with increasing contact line velocity are dissipative effects. For example, wave amplitude against a vertical wall (gravity waves) is dampened not only by viscosity but also by the contact line 'friction' against the wall.

If a wetting layer of lower phase is formed between the upper phase and the container and/or vapor (at temperatures above T_w), then the contact line 'friction' should be different from that for partially wetting conditions found at temperatures below T_w. This layer of the lower phase separates the upper phase from the glass and vapor. The contact line which sweeps up and down is no longer the tri-junction between the upper fluid, lower fluid and the glass phases.

It is known that fully wetting fluids do not have contact angle hysteresis (as the contact angle is zero). We cannot expect the magnitude of the dissipative effects at the contact line to be the same during partial wetting conditions. Young and Davis state that the dissipative effect of contact angle steepening with increased contact line speed is suppressed for cases of fixed contact angle and fixed contact line when the contact line is independent of contact line speed. Full wetting means fixed contact angle. Increased dissipation of the meniscus oscillation is therefore expected for $T < T_w$.

Differentiating the dissipative effect of viscosity from that of contact line 'friction' is possible when experiments are performed with conditions such that the rate of relaxation of the fluid at the solid-liquid-gas junction is much greater than the velocity. Johnson et al could ignore viscosity effects for their experiments because their conditions satisfied the above assumption.

This Experiment

Interfacial free energy, viscosity and density difference all diminish as temperature increases. There is no singularity in these functions. CPW is a first-order wetting transition which should manifest a singularity in Young's equation or in fluid layer thickness. By using the meniscus oscillations and comparing them in the temperature regime about T_w, the wetting

transition should lead to a singularity in the damping behavior of these oscillations. There has been qualitative evidence to indicate there is an effect. To date, there is too little data of sufficient quality to confirm the theory.

There is clear evidence that the damping of the oscillation wave amplitude is greater for $T < T_w$ in the P-I system. This might be attributed to the increased viscosity. Measurements or calculations will need to be made to verify that viscosity is not the sole dissipation mechanism. Alternately, more data can be collected to permit an empirical curve to be drawn showing the damping factor as a function of temperature.

A difference in period of oscillation has been observed between temperatures above and below the T_w. Below T_w the frequency of oscillation is slightly higher. This effect may be due to the increase in interfacial tension as temperature is decreased. There is not enough data to fit curves which could show any more than this.

The observation that initiated the contact line 'friction' concept is a very subtle difference in the rate the period changes as the meniscus oscillates during a drop between the temperatures above and below T_w. At $T < T_w$, the period of oscillation decreases constantly while the amplitude is damped. At $T > T_w$, the period remains constant. Note that this is a different effect than damping with constant fundamental frequency. Measurements will be needed to ensure viscous relaxation of the fluid is more rapid than the contact line velocity. Contact line velocity has been measured to have a maximum of 2.6 mm/sec. This value places it roughly in the non-viscous controlled domain. The observation is not confirmed because to clearly show it, the oscillation data needs to be improved.

Experiment Status to Date

The major delays have been with the tower itself. After a year of struggling to get the video telemetry system working, it now seems to function as it should have. Clearly, the system was defective as received from the manufacturer.

A drop was made just a few days ago where the whole video system was functionally operative. Adjustments are neccesary to permit data collection, but these little problems will be ironed out over the next month. Video from the experiment was received and recorded from prior to the release of the package to the impact point. A picture was received for nearly the whole duration of the drop except when the drag shield encountered a rough spot on the guide rails. This must be repaired soon. As a result, the picture was distorted or briefly interrupted near the end of the drop. From the video recording it was possible to confirm that although the drag shield was

knocked about by hitting the rail, the experiment inside was not affected since it was adequately levitated within. When the drag shield hit the rail very hard, the laser telemetry was knocked out of alignment briefly since it is directly mounted to the top of the drag shield.

From all the drops made this last year, only one (DT 7-87) provided the accelerometer data for both recording methods. The radio telemetered data and the on board Geo-tek system were both working properly such that a comparison between them could finally be made. Since the sampling rates were not equal and not synchronized, a one-for-one correspondence cannot be made. The qualitative match between the waveforms was very good. Quantitatively, only the steady lo-g portion of the drop can be compared. The NASA Lo G Accelerometers cannot provide the unity-g response during the release of the drag shield. Geo-tek data cannot be taken with as high a resolution as the NASA accelerometers can (8 bits versus 24). Both accelerometer data sets showed good agreement of g-level (3×10^{-3} g for both) given the resolution. In order to record the quality of the release acceleration, the Geo-tek was set for a less sensitive scale. As a result, the maximum resolution of lo-g could not be obtained or compared.

During March of 1988, several drops were made to determine the quality of the forces the drag shield sustains during a drop. The results of dropping a high-g and low-g accelerometer have shown that a fault exists on the guide rail and that the experiment package could benefit from a higher levitation point. The dent in the drag shield seems not to be getting worse at present. Results from from these tests and other examinations of Tower data indicated the following courses of action, all of which affect the CPW experiment...

1. The rail is deformed and needs replacement in one location near the bottom of the drop distance. A first attempt at improving it will be to grind the high spots off. If neccesary, the rail section will be replaced. The quality of the low-g and of the laser telemetry is compromised until some repair is made.
2. The thrust pressure needs to be increased. This will produce a higher boost at the start which will cause the package to be raised higher within the drag shield. One consequence of this is the higher anticipated impact forces both on the drag shield and the experiment.
3. For better cushioning at higher thrust, the cushioning material in the catch tube will need upgrading or replacement.
4. Better package balancing will reduce the need for increasing the thrust.

All these facility improvements will be required for the CPW experiment. From the onset (1985), the quality and length of the low-g period of the drop has deteriorated (drop from 3.2 seconds to 2.5 seconds). Longer, bump-free low-g times will

give better results when the analysis of interface oscillations is done.

Processing of the telemetered data is performed under a contract by NTI at MSFC. For the last set of conversions, a delay was encountered since funding for the effort was not readily available, although it was supposed to have been. There is no problem in getting the data converted so long that there is money in the contract for this work and so long as there are no higher priority jobs on their manifests. Further analysis may not be needed so long as the Geo-tek data can be relied upon. The need for radio telemetry will also be eliminated. Only a pressure warning device needs to be added to the drag shield to satisfy safety requirements and eliminated telemetered monitoring of the thruster sphere pressure.

Up to date results and analysis from this experiment was presented at the Microgravity Materials Processing Session at the TMS-AIME Conference in Phoenix Az. in January 1988. A paper was written that describes the experiment results in detail. This paper has been submitted and is expected to be in the Proceedings of the conference and will be a publication. The cost for the trip was covered from this grant. All remaining monies in the grant are to be applied to salary, overhead and benefits for completion of the grant. There is too little money available to extend the grant any further than it is.

This experiment is no longer the only one for the facility. A metals levitation experiment is being developed at Vanderbilt University by Bayuzak et al. A Japanese Astronaut/Scientist, named Mouri, is also performing experiments on the tower in metals casting. Scheduling of drops will now be neccesary among us. Mouri expects 40 drops in the next year, and the CPW experiment will need 5 good ones, where probably 10 will need to be made in order to get the 5 good ones.

Using the NASA Lewis Drop Tower

Due to the never-ending delays, it was suggested at a recent review that the Lewis Drop Tower be used for this experiment. While the experiment could have been initially designed to be performed in that facility, in its present construction, at this late date, it is far to large to be accommodated at the Lewis Tower.

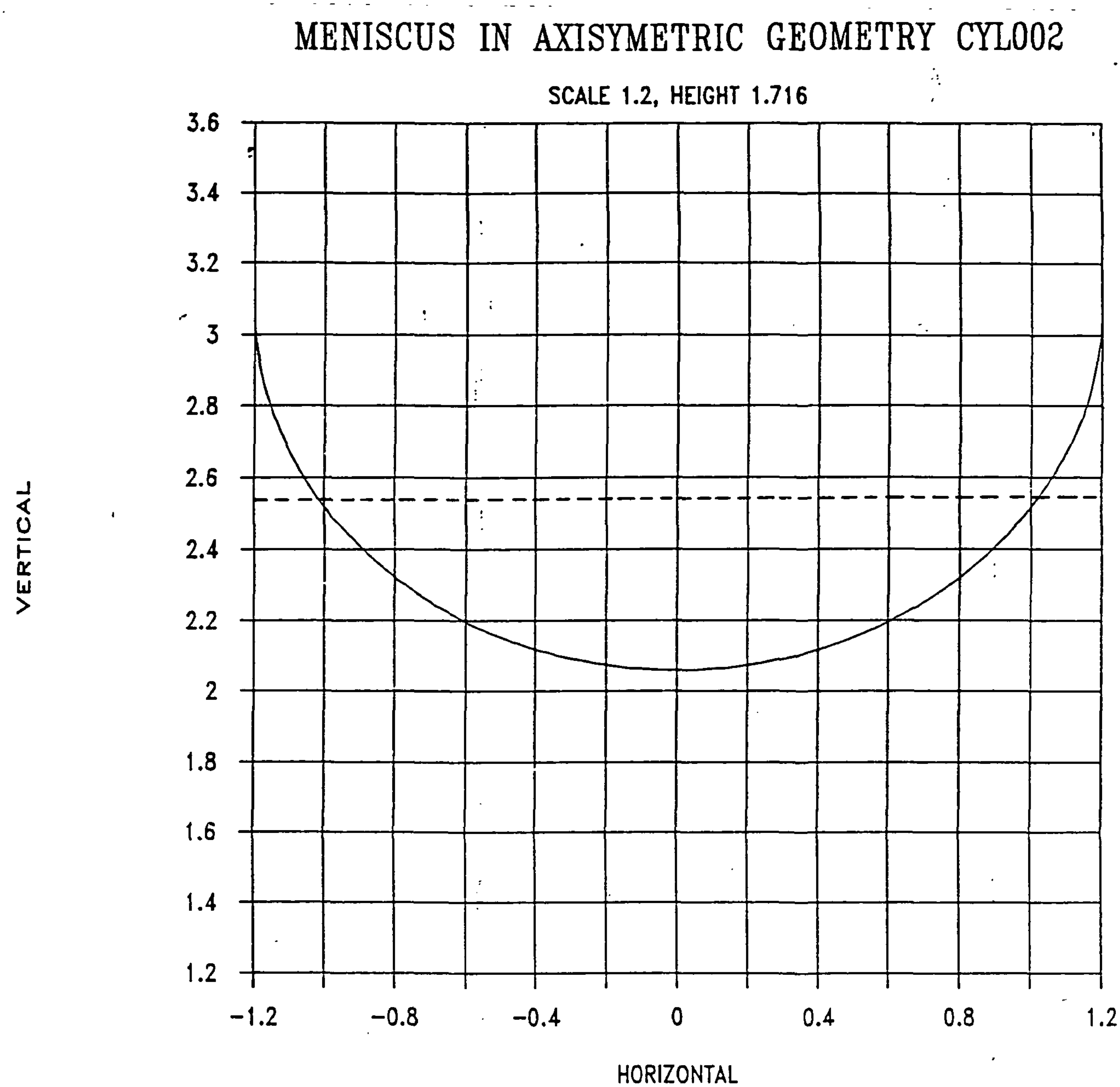
Experimenters at the Lewis facility do have high grade technical support people. The facility is properly maintained. Much of this comes from the high visibility this multi-million dollar facility has. The advantage of the MSFC tower is the large experiment size and weight that can be accommodated. The experiment need not operate in a vacuum environment. Arranging to drop is not a major bureaucratic undertaking. However, sup-

port from NASA for the MSFC Drop Tower is at best weak.

The shape of the drop capsules for the Lewis Tower is also difficult to work within, approximately 2 foot diameter by 10 foot long cylinders are dropped and caught in a bed of foam chips. The g-level at impact is dangerously high (greater than 30 g's) and no on-board video is available during the drop. For these and other reasons, it is not recommended that the Lewis Tower be used for this experiment.

Conclusion

More drops will be made with the new video telemetry system and analysis of this data will follow as the video results arrive. The year extension provides the time to obtain this data and prepare the final science report. The quality of the data will determine how well the wetting transition can be delineated by this experiment technique. So far, only marginal results have been obtained. There will be confirmation of the theory by these experiments if the data quality is as good as is expected.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WATER